

## METHODS

### COMPARISON OF METHODS FOR DETERMINING THE INTENSITY OF SOIL RESPIRATION

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Measurement of the rate of release of carbon dioxide from the surface of the soil (soil respiration) is widely used in practical studies for estimating the biological activity of the soil and one of the indicators of the effect of agronomic, chemical, forest-management, and reclamation measures on the soil.

But the results of the soil respiration studies made with different methods often differ substantially from one another and they cannot be compared directly. The present paper is devoted to a comparative evaluation of the principal methods for determining the intensity of soil respiration in order to show the most reliable and promising of the methods.

One principle lies at the basis of all methods for determining the intensity of soil respiration in use at the present time, namely, that a definite area of the soil surface, which is cleared of vegetation, is isolated from the air of the atmosphere by an isolation chamber (a Lundegardh bell-jar). The carbon dioxide evolved from the soil into the chamber is determined quantitatively by one or another method.

The means for directly determining carbon dioxide can be divided into three basic types, of which there are many modifications.

1. Enrichment method. Introduced by Lundegardh [11]. An isolation chamber is set out on the surface of the soil for a definite period of time and then a sample of air enriched in carbon dioxide is removed from it and the  $\text{CO}_2$  content of the sample is determined.

2. Air current method. Used by Humfeld [8]. Air from the outside is drawn through an isolation chamber continuously at a definite rate. The  $\text{CO}_2$  is determined quantitatively in the air coming from the chamber that is enriched in carbon dioxide.

3. Absorption method. First used to determine the intensity of soil respiration by Bornemann in 1919 [6], and Lundegardh [10]. Immediately before beginning the determination a titrated solution of alkali is placed into a vessel in the isolation chamber to absorb the carbon dioxide evolved from the soil. After a definite period of time the remaining free alkali is measured and the amount of carbon dioxide evolved is calculated.

All these methods and their rather numerous variants and modifications have an empirical basis. For this reason the value of results obtained from their use depends to a great extent on a successful choice of parameters and elements composing the particular method used. These include the volume of the chamber, the ratio of the area of isolated surface to the height of the chamber, the length of time the chamber is in place, the rate at which air is drawn through the chamber, the ratio of the absorption area to the area isolated, the concentration of alkali solution in the absorber, etc.

In evaluating and recommending one method over the others mentioned previously, preference should be given to the one which causes minimum interference with the natural process of  $\text{CO}_2$  evolution from the soil and at the same time permit most precise determination of the carbon dioxide evolved from the isolated surface during the test period.

Of all the factors effecting the movement of carbon dioxide in the upper layer of the soil and its evolution into the atmosphere, the most important is diffusion. The diffusion rate of carbon dioxide from the soil into the atmosphere depends primarily on the intensity of the processes governing the formation and release of  $\text{CO}_2$  into the soil atmosphere and those governing the concentration gradient  $\text{CO}_2$  in the soil and the air of the soil and that of the atmosphere. The diffusion rate of carbon dioxide also depends on temperature, moisture, amount of air in the soil, the size and number of effective (free of water) pores in the surface layers of the soil. The set of conditions that make up a concrete situation determines the rate of evolution of carbon dioxide from the soil. In the development of a method for determining the intensity of soil respiration, the most difficult aspect is the impossibility of keeping the  $\text{CO}_2$  concentration gradient at the initial natural level and consequently also the rate of  $\text{CO}_2$  diffusion.

Hence an evaluation of the method for determining the intensity of  $\text{CO}_2$  evolution from the soil should be based on these premises.

#### Enrichment Method

Use of the enrichment method is inevitably

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associated with a decrease in the concentration gradient, since it involves the very principle of the method, but the quantitative expression of this decrease depends on the parameters of the isolation chamber and the length of time it is kept on the soil. In practice the decrease in the  $\text{CO}_2$  concentration gradient can be kept to such a low value that the error caused in determining the intensity of the soil respiration can be neglected. For purposes of illustration we shall use values for three isolation chambers used in practical studies: the Lundegardh bell-jar, the Makarov dome, and the aluminum pan used by the forestry laboratory. Adopting conventional values for the initial  $\text{CO}_2$  concentration gradient but based on actual values, let us compute the average relative decrease in the concentration gradient.<sup>1</sup>

We adopt the following conditions:

1. The concentration of  $\text{CO}_2$  in the soil atmosphere of the upper soil layer is equal to 9.8 mg/liter (0.5%).

2. The concentration of  $\text{CO}_2$  in the air layer near the ground is 0.98 mg/liter (0.05%).

Hence the initial concentration gradient for  $\text{CO}_2$  is equal to 8.82 mg/liter.

3. The intensity of  $\text{CO}_2$  evolved from the soil into the atmosphere is 4 mg/dm<sup>2</sup> hr (corresponding to 4 kg/ha hr).

Let us use the following simple formulas to make our computation:

$$mxmxmxmxmxmxmx$$

where  $u$  is the intensity of  $\text{CO}_2$  evolution from the soil (mg/dm<sup>2</sup> per hour);  $s$  is the area of the isolation chamber in dm<sup>2</sup>;  $v$  is the volume of the isolation chamber in liter;  $t$  is the length of time the isolation chamber is in place on the soil, in one hr;  $a$  is the increase in  $\text{CO}_2$  concentration, equal to the decrease in gradient during time  $t$ ;  $g$  is the initial concentration gradient for  $\text{CO}_2$  (mg/liter); and  $c$  is the mean relative decrease in the concentration gradient, which is equal to the mean relative decrease in the rate of  $\text{CO}_2$  diffusion from the soil during time  $t$ .

If the isolation chamber has the shape of a

parallelepiped or a cylinder, formula (1) may be simplified to the following:

$$a = \frac{ut}{h},$$

where  $h$  is the height of the chamber in dm, or in other words, the decrease in the  $\text{CO}_2$  concentration gradient is directly proportional to the intensity of  $\text{CO}_2$  evolved from the soil and the length of time the isolation chamber is in place and inversely proportional to the height of the isolation chamber.

From the formulas given and the data computed in Table 1 it follows that the isolation chamber for determining the intensity of soil respiration by the enrichment method should be rather high — not less than 25 cm. When an isolation chamber of this sort is used and it is kept in place no more than 30 minutes, the relative error of determination of the respiration intensity caused by the decrease in the  $\text{CO}_2$  concentration gradient does not exceed 5% for average conditions, i. e., it falls within the limits of accuracy of the measurements. At high intensity of soil respiration it is completely possible to hold the relative error within the same limit by correspondingly reducing the time that the isolation chamber is in place on the soil.

The use of small isolation chambers with a large isolated area such as, for example, the Lundegardh bell-jar, should be discontinued, since in this case even with a short period in place a considerable error is unavoidable and the results of the determination of respiration intensity will be much too low.

The best modification of the enrichment method, although it involves the use of rather cumbersome equipment, is the modification of Makarov [2]. According to this method, an isolation chamber (dome) with a volume of 83 dm<sup>3</sup> (28x58x51 cm) is set in place for a period of 30 minutes on the soil, and then a 2-liter sample of air enriched in carbon dioxide is removed from the chamber with an aspirator. After its removal from the dome, the air sample is passed through a titrated solution of baryta placed in a Rickhter absorber. After passing the sample through, the remaining free alkali is determined by titration with acid in the presence of phenolphthalein. The quantity of baryta combined with carbon dioxide is computed from the difference between the amount of baryta taken for use as an absorbent and the amount that remains free. At the time the dome is set in place on the soil the initial concentration of  $\text{CO}_2$  before the determination is determined by passing two liters of air from the atmospheric layer near the ground at a height of 30–40 cm through the baryta. The quantity of baryta consumed by the  $\text{CO}_2$  evolved from the soil during the determination period is computed from the difference in consumption of baryta by  $\text{CO}_2$  before and after the period during which the dome is in place.

As indicated by the data given in Table 1, the relative error from the decrease in the  $\text{CO}_2$  concentration gradient using the Makarov dome is very small (about 2% for a half-hour

<sup>1</sup>When the isolation chamber is first set in place on the soil, the  $\text{CO}_2$  concentration gradient is at its initial value and consequently the decrease in gradient is equal to zero. The increase in the concentration of  $\text{CO}_2$  at the end of the period when the chamber is in place is equal to the decrease in the concentration gradient. The final concentration gradient, divided by 2, will give, in first approximation, the value of the mean decrease in gradient during the experimental period. This value, expressed as a percent of the initial  $\text{CO}_2$  concentration gradient, gives the value for the mean relative decrease in the concentration gradient, which is also equal to the relative decrease in the rate of  $\text{CO}_2$  diffusion from the soil into the atmosphere.

determination) and it may be ignored.

Theoretically there is still one "error" in the Makarov method. When the sample is removed from the dome it is necessarily replaced by an equal volume of air from the upper layer of the soil. Since, in comparison with the atmospheric air, the soil air is considerably richer in carbon dioxide, then the air that enters the chamber from the soil increases its  $\text{CO}_2$  concentration. When the isolation chamber is large, the error in the determination of the intensity of soil respiration from "contamination" of the sample by the soil air is very slight. Under the conditions adopted here it is about 1.5%, and since this error is in the direction opposite to the decreased intensity of  $\text{CO}_2$  diffusion from the soil caused by the decrease in the concentration gradient, a certain mutual compensation results.

be a disturbance of the  $\text{CO}_2$  concentration gradient but the wind stream created will withdraw additional soil air through the pores into the chamber and the natural respiration intensity of the soil will be disturbed. In addition, when air is drawn through the chamber, the atmospheric pressure in the chamber is altered; a certain rarefaction is created without which it would be impossible under isothermal conditions to create a continuous stream of air. The pressure gradient created also causes additional evolution of soil air into the chamber.

Thus in this method, even when the velocity of the air current is adjusted to the rate of  $\text{CO}_2$  evolution from the soil, the determination will include, in addition to the carbon dioxide evolved during soil respiration, a certain additional amount withdrawn together with the soil atmosphere.

Table 1

Mean relative decrease in the  $\text{CO}_2$  concentration gradient

Designation and dimensions of the isolation chamber		Length of time chamber is in place, min.			
		10	20	30	60
Lundegardh bell-jar, Cone	$s=7.5$ $v=2.3$	12.3	24.6	36.9	73.8
Makarov dome, Parallelepiped	$s=16.0$ $v=83$ $h=5.2$				
Aluminum pan	$s=15.2$	0.72	1.45	2.17	4.34
Cylinder	$v=38$ $h=2.5$				
		1.5	3.0	4.5	9.0

Note: Comma represents decimal point.

When the dimensions of the isolation chamber are less than indicated in the Makarov method, it is desirable to eliminate the "contamination" of the air in the chamber by the soil air. This is easily achieved by placing the open lower bottle of the aspirator inside the isolation chamber before the determination of respiration begins. The bottle should be placed on a wire support and not directly on the soil and instead of water a saturated table salt solution should be placed in the aspirator. Both aspirator bottles are joined by a rubber tube running through a glass tube hermetically sealed to the top of the chamber. When the air sample is sucked from the chamber, the volume of air removed will be replaced by an equal volume of liquid entering the lower bottle of the aspirator, and additional movement of soil air into the chamber will be eliminated.

#### Air Current Method

In this method the carbon dioxide evolved from the soil is withdrawn from the chamber together with the air passed through the chamber. When the air stream is moving fast enough the evolution of carbon dioxide from the soil will be practically equal to the rate of the removal from the chamber. In this case there will not

Gorbunov, Shkolnik and Morozova [1], in checking the air current method (erroneously ascribing its introduction to Lundegardh), came to the conclusion that the quantity of carbon dioxide evolved depends not so much on the area of soil isolated as on the volume of air drawn through the chamber. This conclusion confirms the considerations outlined previously above concerning the direction of the disturbance of the natural respiration process when air is passed through the isolation chamber. Hence use of the air current method for determining the intensity of soil respiration should be avoided, even though it is used in practical studies, particularly abroad [8, 9, 14, etc.]. And the method used by Wallis and Wilde bears only a general resemblance to the air current method. In their method air is withdrawn from the soil through a glass funnel or cylinder inserted in the soil using a vacuum pump. The carbon dioxide withdrawn by this means is no indication of soil respiration.

#### Absorption Method

As compared with the enrichment method, the absorption method is based on the opposite principle — that of removal of the carbon dioxide evolved from the soil into the chamber

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during the determination period. Depending on how well the rate of  $\text{CO}_2$  evolution into the chamber corresponds to its absorption by the alkali solution, the carbon dioxide concentration gradient will be altered or maintained at its initial level. It is theoretically possible to balance the soil area isolated and the area of the absorber vessel in such a way that the quantity of carbon dioxide evolved from the soil and the quantity absorbed in unit time will be equal. Consequently, from the theoretical point of view, the absorption method can be considered quite promising.

The most important factors governing the correctness and reliability of soil respiration intensity determinations by this method are the ratio of the area isolated to the absorbing area, the concentration and quantity of the absorbing alkali solution, the temperature, and the size of the chamber.

After initial methodological work by Bornemann [6], Lundegardh [10] and Meinek [13, 19], this method was used by Walter [15], Haber [7] and others. In the Soviet Union a modification of this method was worked out by Shtatanov [4]. Because of its simplicity it found wide use in practical studies. But data obtained by this method in its original form most of the time were much too low.

A comparison of the results of soil respiration intensity determination obtained for various soils by Makarov showed that the values obtained by the Shtatanov method were much lower than those obtained by the Makarov method. The same verification was later made by Shtatanov. He came to the conclusion that the results obtained by the two methods were close to one another [5]. The differences in the results of verification are apparently due to the fact that Makarov used the Shtatanov method in the first modification [4] and Shtatanov in a form modified after additional methodological work [5].

It should be noted that many investigators using the absorption method did methodological work and introduced changes but none of the modifications in the literature can be considered sufficiently well worked out.

Nevertheless, as mentioned previously, the absorption method should be considered promising and its development would be very desirable. To support this conclusion we present the following considerations. In one Shtatanov's experiments<sup>2</sup> there was established a clear dependence between the surface area of the absorber and the quantity of carbon dioxide freely moving from the atmosphere absorbed in unit time. For a 0.2 N solution of sodium hydroxide, the rate of  $\text{CO}_2$  absorption averaged 0.25 mg  $\text{CO}_2$   $\text{cm}^2$ /hr. Assuming the mean intensity of  $\text{CO}_2$  evolution from the soil to be 0.04 mg  $\text{CO}_2$   $\text{cm}^2$ /hr (corresponding to 4 kg/ha hr), it is possible to calculate a

relationship of isolation chamber and absorber areas at which the initial  $\text{CO}_2$  concentration gradient will not be disturbed during the determination. For the assumed conditions this ratio is equal to 6; in other words, to preserve the initial value of the concentration gradient, the absorption surface with average intensity of soil respiration should constitute no less than 16–17% of the area of soil isolated. When the intensity of soil respiration is higher, the absorption surface should be correspondingly increased. Thus, when the intensity of respiration is 0.08 mg/ $\text{cm}^2$ /hr, the absorption surface should be increased to 32–35%.

In a later modification of the Shtatanov method an isolation chamber was used which was shaped like a truncated cone with an isolated area of 200  $\text{cm}^2$  and the absorber was a Betri dish 10 cm in diameter which amounted to 36% of the area isolated.

In the first modification of the Shtatanov method insufficient attention was paid to the relationship of areas of the chamber and the absorber. The area of the absorber was small and the concentration of the alkali solution was twice as small as it should have been, and this combination of circumstances led to results that fluctuated and were largely too low. The recent modification of the Shtatanov method apparently is free of these deficiencies to a considerable extent.

It is necessary to emphasize one theoretical feature of the absorption method. Increasing the area of the absorber even to the maximum limits, making it almost as large as the isolated area, can create comparatively little increase in the initial  $\text{CO}_2$  concentration gradient, and consequently a small increase in the rate of carbon dioxide diffusion from the soil.

If we return to the quantities adopted previously, where the  $\text{CO}_2$  concentration in the soil atmosphere in the upper layer was equal to 9.8 mg/liter and that in the atmospheric layer close to the ground was 0.98 mg/liter, then with maximum rate of  $\text{CO}_2$  absorption by the alkali in the absorber, its concentration in the chamber will equal approximately zero. Consequently the initial  $\text{CO}_2$  concentration gradient increases from 8.82 to 9.8 mg/liter. The maximum error in determination of respiration intensity amounts to

$$\frac{(9.8 - 8.82) \cdot 100}{8.82} = 11\%.$$

In practice the error will be lower, since the  $\text{CO}_2$  content in the air of the chamber cannot be reduced to zero. From the foregoing computations given it follows that equipment designed for high respiration intensities can be used successfully also with low respiration intensity. The relative error in this case will be within allowable limits.

The analysis of methods for determining the intensity of soil respiration presented not only does not exclude but rather emphasizes the necessity of developing more promising methods, particularly the absorption method. The principles on which the critique of the

<sup>2</sup>Personal communication.

method was based can be the basis for further methodological studies.

Received March 5, 1962

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